

optical scattering and microstructure of ion beam deposited metal coatings

G.A. Al-Jumaily, N. A. Raouf, S. M. Edlou* and J. Simons*

Jet Propulsion Laboratory, California Institute of Technology
4800 Oak Grove Drive, Pasadena, CA 91109

ABSTRACT

Thin films of gold and platinum have been deposited onto super-polished fused silica substrates using thermal evaporation, ion assisted deposition (IAD), sputtering and ion assisted sputtering. The influence of ion beam flux, coating material and deposition rate on the films microroughness have been investigated. Coatings of gold and platinum have been bombarded with low energy (10-20 eV) Ar ions from an electron cyclotron resonance (ECR) ion source during deposition. Short range surface microroughness of coated surfaces has been examined using scanning tunneling microscopy (STM) and atomic force microscopy (AFM), while long range surface microroughness has been characterized using an angle resolved optical scatterometer. Results indicate that bombardment with low energy ions causes significant reduction in microroughness of metal coatings.

2. INTRODUCTION

Metal coatings are widely used as wide-band reflectors for mirror applications. Aluminum (Al) and silver (Ag) are the most commonly used materials. Silver has the highest reflectance in the visible and IR regions. Its usefulness has been limited by poor environmental instability. Although Al is more stable than Ag, it suffers from lower reflectivity in the visible. Gold (Au) coatings are very stable environmentally and have very high reflectivity in the IR. Evaporated Au coatings have very low reflectance in the UV-visible region of the spectrum in addition to poor adhesion.

Improving the durability and reducing optical scattering are major concerns for space and ground based astronomy. Scattering prohibits the telescope's ability to resolve faint planets nearby much larger and brighter stars. Scattering in metal coated surfaces is caused by the surface roughness and optical figure of the uncoated substrate and the microstructure of the coatings. Surface roughness of the substrate can be minimized by using a single crystal substrate such as silicon or super polished amorphous materials such

*Barr Associates, Inc., 21 Liberty Way, Westford, MA 01886

as fused silica or Zerodur. Coating microroughness is caused by the columnar growth of thin films. Ion

beam deposition processes have been used to modify the columnar growth process and reduce the microroughness of thin films. [1 -6]

In an earlier publication we have shown that applying ion beams to the deposition processes of thin films have greatly influenced the microstructure of metal coatings. [2-6] It has been noted that high energy (300-500 eV) ion bombardment of metal coatings increases the microroughness. In this paper we discuss the influence of low energy (10-20 eV) ion bombardment on the microroughness of Au and Pt coatings deposited using sputtering, and ion assisted sputtering.

3. SAMPLE PREPARATION

Coatings approximately 100 nm thick, were deposited in a 60 cm box coater at Barr Associates. The coating system was cryogenically pumped to a working pressure of 2×10^{-6} Torr. The substrates were not heated and bias voltage was not applied to the substrate holder. The deposition system was equipped with an ECR ion source, a 7.5 cm dc sputtering source and a resistance heated evaporation source. Ultra high purity argon was used in the ion source at a pressure of 2×10^{-4} Torr. For sputtering the total pressure was 1×10^{-3} Torr. Deposition rates and film thickness were monitored using a crystal rate monitor as well as an optical monitor.

Gold and platinum films were deposited using sputtering and ion assisted sputtering. In the ion assisted sputtering case, coatings were bombarded with low energy Ar ions from an ECR source. The superpolished fused silica substrates were ion pre-cleaned prior to coating.

4. CHARACTERIZATION TECHNIQUES

The microroughness of coated and uncoated substrates have been characterized using the following techniques:

4.1. Spectral Reflectance

Spectral reflectance of the coated substrates was measured using a dual beam spectrophotometer with a reflectance attachment. The measurement range was 500 to 2000 nm. A special emphasis has been given to changes in the reflectivity near the absorption edge.

4.2. Angle-Resolved Optical Scatterometer

Gold and platinum coated superpolished fused silica substrates have been examined using an angle resolved optical scatterometer (AROS) at Sandia Systems. [6] The AROS system uses a He-Ne laser ($\lambda = 632.8 \text{ nm}$) as a source. The laser beam is focused onto the detector. Hence measurement is done in the far field. The scattered light intensity is measured in the plane of incidence using a photomultiplier tube. The spatial wavelength range of the system determined by the system geometry and the source wavelength is 0.7 to $72 \mu\text{m}$. A detailed description of the system can be found elsewhere. [2-4]

The scattered light characteristics of the surface can be presented as the bi-directional reflectance distribution function (BRDF) or the power spectral density (PSD). A value for the RMS roughness is obtained by integrating the PSD curve.

4.3. Scanning Tunneling and Atomic Force Microscopy

The scanning tunneling microscopy (STM) and atomic force microscopy (AFM) were used to examine the surface microroughness of uncoated and coated substrates. The STM technique profiles surfaces using a tungsten stylus which stays close to the surface and is moved across the surface while keeping the tunneling current constant. Therefore the surface has to be conductive. The AFM is similar to the STM except the probe touches the surface and the repulsive force is held constant.

5. RESULTS

Coatings of Au and Pt have been deposited to examine the effects of deposition process parameters on the microstructure of coatings. Results for both materials are discussed below:

5.1. Uncoated Substrates

The microroughness of the uncoated, superpolished fused silica substrates has been examined using an AFM. Figure 1A is a micrograph of the fused silica over an area of $2 \mu\text{m} \times 2 \mu\text{m}$. The calculated RMS roughness is 2.4 Å. The same sample has been examined over an area of $10 \mu\text{m} \times 10 \mu\text{m}$. Results are shown in Fig. 1B. The calculated RMS roughness is 2.0 Å which is very close to that of the smaller scan.

5.2. Gold Coatings

We have examined the effects of applying a thin metallic coating of Au on the roughness of superpolished fused silica. Figure 2 represents two STM micrographs of Au

1. *Y. enterocolitica* serotype 4/O:3, which is the most common serotype in the United States, was isolated from 100% of the samples.

Values of RMS roughness for Au coatings calculate] from scatter measurement and STM “.

Sample	RMS from Scattering (Å)	RMS from STM (Å)
Sputtered Au	14.4	10.0
Sputtered +1Al) Au	9.9	5.5

The reflectivity of Au coatings has been characterized. Figure 3 illustrates the reflectance of sputtered and Al O-sputtered Au coatings. It is concluded from Fig. 3 that no reduction in the reflectivity of sputtered Au coatings as a result of bombardment with low energy ions.

The microroughness of Pt coatings deposited using sputtering and IAS onto super polished fused silica have been examined. Figure 4 represents two STM micrographs of Pt coatings. The RMS roughness values for the 2X2 μ m and 10X10 μ m micrographs are 5.5 Å and 5.1 Å, respectively. Comparing Figures 2 and 4, it can be concluded that the grain size of the Pt film is much smaller than the Au coating.

The surface roughness and microstructure of Au and Pt coatings deposited onto superpolished fused silica substrates using ion beam deposition processes have been examined. Results presented above indicate that ion beam deposition techniques (sputtering and IAl) sputtering) introduce significant changes to the coating microstructure. In general, ion bombardment causes significant reduction in the surface microroughness compared to thermally evaporated coatings. Sputtered Au films have shown lower surface microroughness than evaporate] and I Al) coatings. However, increased bombardment with high energy ions causes an increase in optical scattering and related surface roughness. We have demonstared that bombardemnt with low energy ions from an ECR source does not cause an icrease in the. surface roughness of Au coatings.

Platinum coatings deposited using sputtering and ion assisted sputtering have lower surface roughness as compared with gold films. Unlike Au coatings, IAS Pt coatings did not show a significant increase in microroughness as a result of ion bombardment. The difference in the way ion bombardment affected the microstructure of Au and Pt can be explained by referring to the zone models of Movchan and Demchishin [7] and Thornton [8]. The melting temperature of Pt is 1772 compared to 1064°C for Au. The transition temperature going from zone I to zone II occurs at about $0.35 T_m$, where T_m is the melting temperature (in Kelvin) of the metal. For Pt and Au the values of the transition temperatures are 443 and 195°C, respectively. The difference in the values of the transition temperatures indicate that low energy is needed to cause significant changes in the microstructure of Au, while much higher energy will be needed to cause similar changes in Pt.

7. CONCLUSIONS

Optical scattering and microstructure of Au and Pt coatings deposited using ion beam processes have been examined. Several characterization techniques have been employed to examine the effects of deposition process parameters on the microroughness of coatings. Results indicate that sputtered Au coatings bombarded during deposition with low energy ions do not exhibit an increase in optical scattering. We have also demonstrated that Pt coatings are smoother than Au coatings deposited under the same conditions.

8. ACKNOWLEDGMENTS

The authors acknowledge Mark Anderson for his help on the AFM and STM measurements, Scott Wilson and Rod Jacobson of Sandia Systems for optical scatter characterization.

This work has been carried out by the Jet Propulsion Laboratory, California Institute of Technology under a contract with the National Aeronautics and Space Administration.

9. REFERENCES

1. J. M. E. Harper, J. J. Coumo, R. I. Gambino, and H. R. Kaufman, in *Ion Bombardment of Surfaces: Fundamentals and Applications*, G. Auciello, and R. Kelly, Eds. (Elsevier, Amsterdam, 1982) p. 745.
2. G. A. Al-Jumaily, J. J. McNally, J. R. McNeil, and W. C. Herrmann, Jr., "Effects of Ion Assisted Deposition on the optical Scatter and Surface Microstructure of Thin Films," *J. Vac. Sci. Technol.* A3, 651-655 (1985).

Use of type is not to be confused with the use of type in the text. Type is not to be used for the text, but for the text only.

3. G. A. Al-Jumaily, S. R. Wilson, J. J. McNally, J. R. McNeil, J. M. Bennett, and I. Hurt, "Influence of Metal Films on the optical Scatter and Related Microstructure of Coated Surfaces", Appl. opt. **25**, 3631-3634 (1986).
4. G. A. Al-Jumaily, "Influence of Metal Films on the Optical Scatter and Related Microstructure of Coated Surfaces", Ph.D. dissertation (University of New Mexico, Albuquerque, NM (1986).
5. D. R. Coulter, N. A. Raouf and G. A. Al-Jumaily, "Microstructure of Metal Coatings. Deposited Using Ion Beam Deposition Processes," Proceedings of the Space optics and Astrophysics Meeting, optical Society of America 1991.
6. R. D. Jacobson, S. R. Wilson, G. A. Al-Jumaily, J. R. McNeil, J. M. Bennett, and Lars Mat tsson, "Microstructure Characterization by Angle-Resolved Scatter and Comparison to Measurements Made by Other Techniques", Appl. opt. **31**, 1426-1435 (1992).
7. B. A. Movchan and A. V. Demchishin, "Study of the structure and properties of thick" condensates of nickel, titanium, tungsten, aluminum oxide and zirconium dioxide," Fiz. Met. Metalloved. **28**, 653-660 (1969).
8. J. A. Thornton, "Influence of apparatus geometry and deposition conditions on the structure and topography of thick sputtered coatings, " J. Vat. Sci. Technol. **11**, 666-670 (1974).

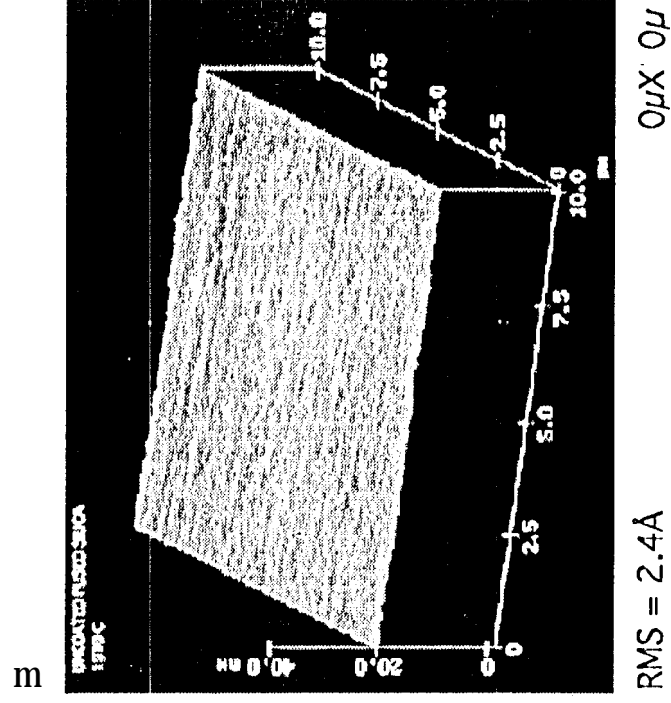
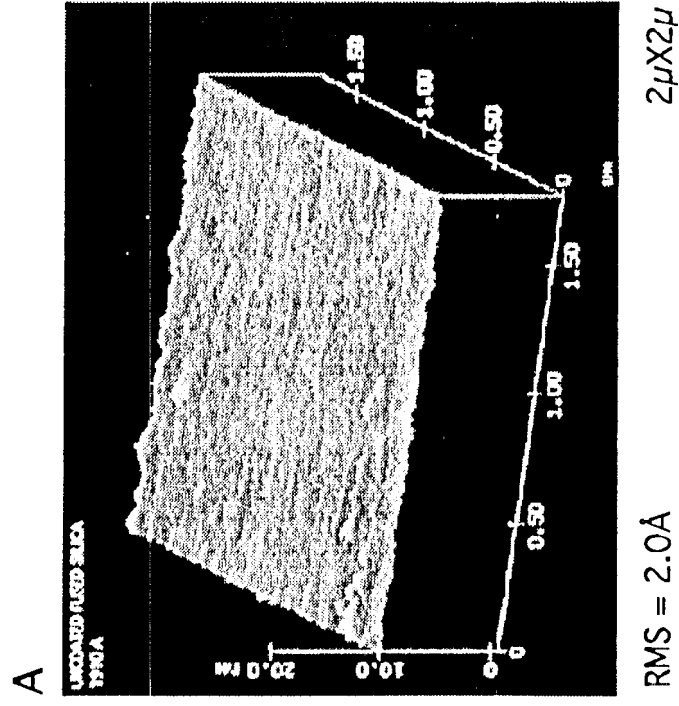
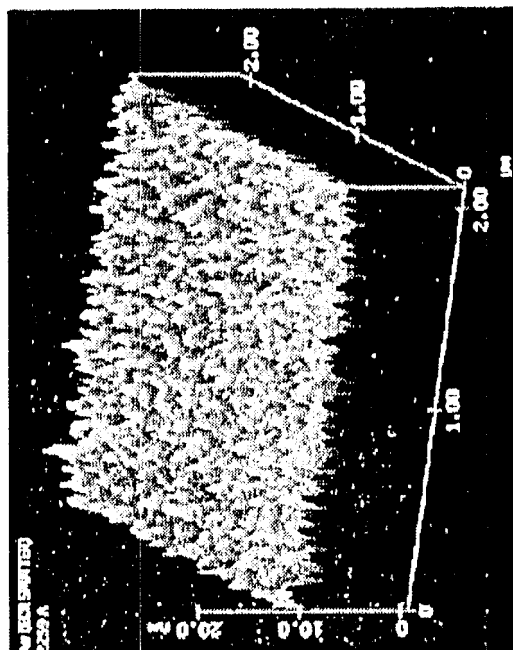
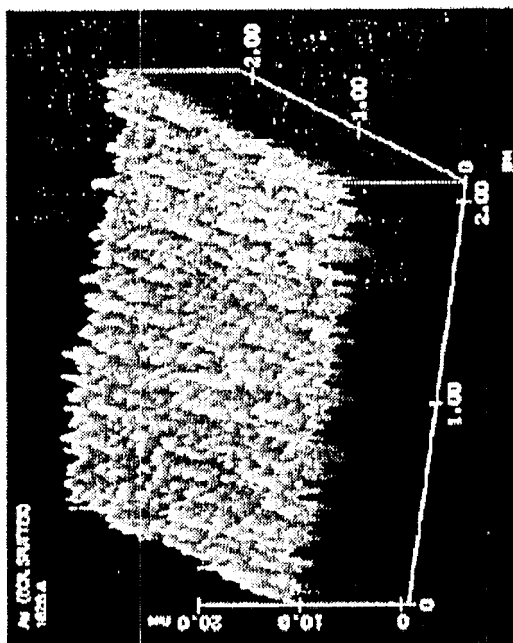


Figure 1. Microstructure of uncoated superpolished fused silica

not beyond the coverage of the patent.



RMS = 8.8 Å sputtered + IAD



RMS = 11.0 Å sputtered

Figure 2. Effects of IAD on the microstructure of sputtered gold

